An effect of synthesis parameters on structural properties of AlN thin films deposited on metal substrates

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Abstract— **AlN thin film was prepared over different metal substrates using DC sputtering at various sputtering parameters. The XRD spectra revealed the presence of mixed (cubic and hexagonal) phases for all samples other than samples prepared at 300W with Ar:N_2 gas ratio of 14:6. The intensities of cubic phases observed at copper (Cu) substrates increased drastically with high sputtering power and N_2 gas flow. Low intensive peak was observed at gas mixer ratio of 14:6. N_2 flow and sputtering power influenced the crystallinity of the AlN thin film with respect to the substrates. Mixed residual stress (compressive and tensile) was observed for all samples and high values were observed at 300W sputtering power at low N_2 gas flow. Crystallite size of AlN thin film varied with respect to sputtering power, gas flow ratio and also substrates. AlN thin film prepared at 250 W showed high dislocation density at high N_2 gas flow ratio. Atomic force microscope results showed rough surface for AlN thin film coated over Al substrates and increased high value was observed at high N_2 gas flow ratio. The particle size of the AlN thin films increased with N_2 gas flow increased with respect to sputtering power and high value was observed with Al substrates.**

Keywords- **AlN; thin film; structural parameters; metal substrates; particle size**

I. INTRODUCTION

Since the high thermal stability of Mo - Al_2O_3 cermet at high operating temperature in vacuum (450 -500 ºC), it is desirable for solar collector tubes for solar thermal electricity applications. Even though, solar absorptance of 0.96 with emittance of 0.16 was observed, the deposition rate is low [1], cost of deposition using planar magnetron sputtering is much higher when compared to reactively sputtered compounds such as stainless steel–carbon (SS–C) [2,3] and Al–N cermet solar coatings [4-7]. Hence Mo - Al_2O_3 is considered more expensive than SS–C and Al–N cermet solar coatings which are also produced using a commercial-scale cylindrical dc sputtering coater. Among these, Aluminum nitride (AlN) has generated much interest due to its unique properties of wide band gap of 6.2 eV, high thermal conductivity (320Wm^{-1} K^{-1}) [8], low thermal expansion coefficient, high chemical and thermal stabilities, high breakdown dielectric strength, and high surface acoustic wave velocity [9-11]. So far, a variety of deposition methods have been reported for AlN synthesis such as reactive sputtering [12], reactive evaporation [13] metal-organic chemical vapor deposition (MOCVD) [14], laser-molecular beam epitaxy [15], pulsed laser deposition (PLD) [16], arc discharge method [17], and chloride-assisted chemical vapor deposition [18]. Among these, reactive sputtering is relatively good and low cost method for the preparation of AlN thin films. The growth condition is an important which influences the structural and optical properties of the film considerably.

Few research reports showed the influence of deposition parameters on the orientation of the AlN films, in which a general guideline promoting a well c-axis oriented thin film was reported. The sputtering pressure plays an important role in the growth of preferred AlN and reported that the preferred orientation of the AlN film changed from (1 0 0) to (0 0 0 2) with decreased sputtering pressure [19–23] and also reported in a decrease of the FWHM of (0 0 0 2) rocking curve [24]. However, it is necessary to understand the influence of other synthesis parameter on the film properties. Iriarte et. al. reported the influence of different substrates on the properties of c-axis oriented AlN thin films [25]. Based on the application, AlN films have been deposited on various substrates such as tungsten [26], sapphire [27] and diamond [28] substrates and reported their properties. This work focuses on the deposition of thin AlN films on different metal substrates (Cu, Al) and study the influence of growth condition such as sputtering power, gas

flow rate and also substrates. The selection of substrates is based on the material used for solar thermal as well as electrical applications. The structural properties such as crystallite size, dislocation density, internal stress, etc., of all prepared samples are also reported here.

II. EXPERIMENTAL METHODS

AlN thin films were deposited on different metal substrates (Al and Cu) using Al (99.99% purity) target (3 inch in diameter and 4 mm in thickness) by DC sputtering (Edwards make, Model-Auto 500). The chamber was initially pumped down to high vacuum 8.2 x 10^{-6} mbar. All AlN thin films were coated at chamber pressure of 8.2 x 10^{-3}. High pure Ar (99.999%) and N_2 (99.999%) were used for AlN coatings. All thin films were coated for three different Ar and N_2 gas mixture ratio (16:4 – 80:20, 14:6 - 70:30 and 13:7 – 65:35) at two different sputtering power (250 and 300 W). The total flow rate is maintained as 20 sccm. The substrates were cleaned by rinsing in ultrasonic bath of acetone and isopropyl alcohol. All AlN thin films were coated at room temperature and the thickness of the film was 800 nm measured for all prepared films by digital thickness monitor. The deposition rate was varied form 0.42 Å / sec to 1.5 Å / sec. In order to remove the surface oxidation of the target, pre-sputtering was carried out for 5 min before starting deposition at Ar pressure of 3.2 x 10^{-3}. To get the uniform thickness, rotary drive system was used and 25 RPM was fixed for all AlN film coatings. The distance between the substrates to target was fixed as 7 cm for all depositions.

The crystalline nature of the as-grown AlN thin films for all samples was investigated by using a high resolution X-ray diffraction (HRXRD, X'pert-PRO, Philips, Netherlands). A CuKα (λ = 1.54056 Å) source was used, with a scanning range between 2θ = 32° and 70°. This range has been selected because most of AlN peaks were observed between this ranges. The surface morphology of the prepared samples was tested by the atomic force microscope (AFM) and the results are reported here.

III. RESULTS AND DISCUSSION

A. *Peak Intensity and Position Analysis*

The X-ray diffraction studies for all prepared thin films are carried out and observed as mixed phases (cubic and hexagonal) with respect to the substrates and also sputtering parameters. The XRD spectra show in Fig. 1 reveals the diffracted profile of AlN thin film prepared at 250 W in different gas ratio. As observed at 200W, the Cu substrates supports the growth of (200) oriented Cubic AlN. But the intensities of the peak are different from the film deposited at 200W. As the N_2 flow rate increases, the intensity of (200) oriented phase decreases and hexagonal phase starts to grow. In addition, a new intensive (200) peak is also observed at 42.86 ° instead of (200) peak observed at 45.36° when the film coated at Ar:N_2 gas ratio of 13:7.

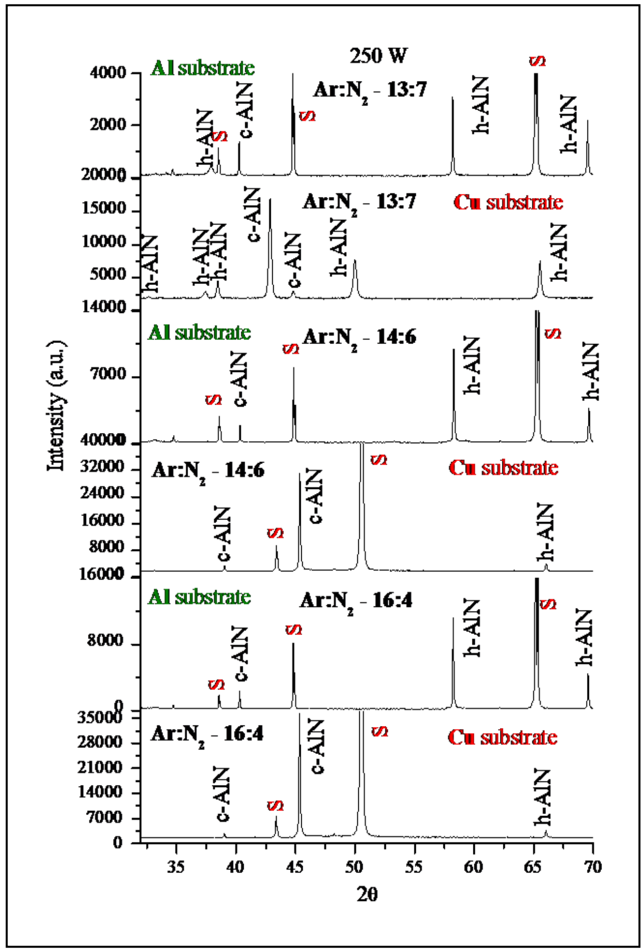


Figure 1. XRD spectra of AlN thin film prepared at 250 W on various substrates and gas flow ratio

It is also observed that cubic phase are nominated but few hexagonal phases are also exist along with cubic phases. Moreover, the (200) peak position shift towards lower 2θ as the N_2 gas ratio increases. But it is also noticed that the peak related to cubic phase observed at 39.02 ° disappear and new hexagonal phases are indexed in AlN thin film prepared at gas ratio of 13:7. On consideration of Al substrates, hexagonal phases are dominated one than cubical phase. In addition, the intensity of peaks decreases as the N_2 gas flow increases ie AlN thin film prepared at 16:4 gas ratio shows higher intensity than prepared at 13:7 gas ratio. The cubic phase also observed on this Al substrates and their intensity decreases as the N2 flow rate increases. It could also be observed that the peak position also shifted towards higher 2θ as with N_2 gas flow increases with respective to observed phases. Overall the crystal orientation as well as the crystallinity of the AlN films for various sputtering parameters depends on the substrates used [29]. Fig. 2

shows the XRD spectra of AlN thin film prepared at 300 W for different gas mixture ratio and observed that no cubic

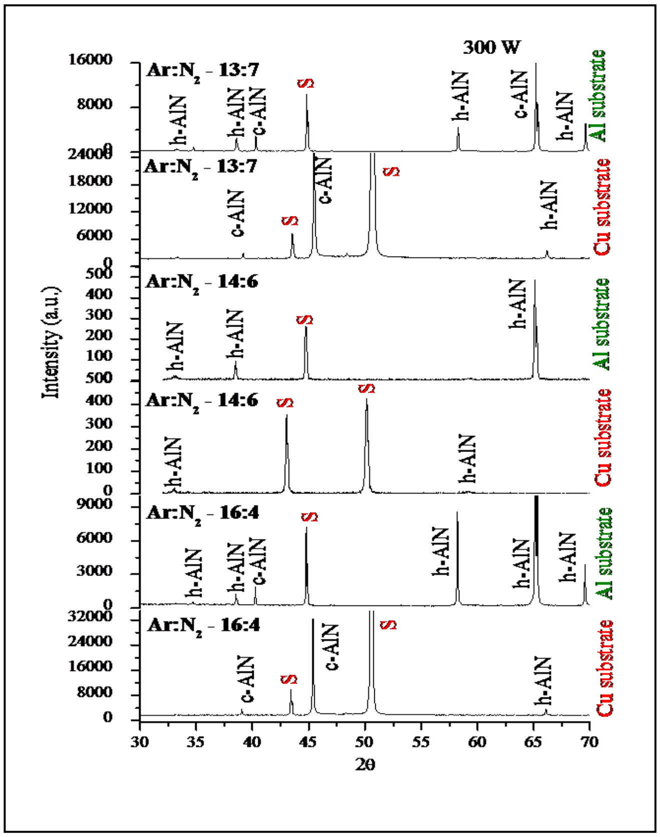


Figure 2. XRD spectra of AlN thin film prepared at 300 W on various substrates and gas flow ratio

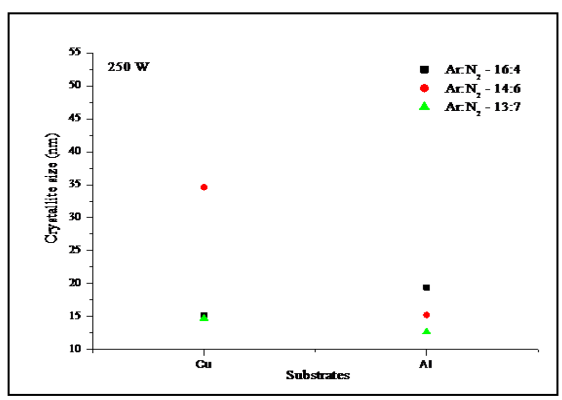
phases are identified from AlN thin film coated on Cu substrates at gas mixture ratio of 14:6. The observed intensity of the hexagonal peaks is low at this gas mixture (14:6) compared to other gas mixture ratio for both Al and Cu substrates. The intensity reduction and peak shift are also could be observed as the N_2 gas flow increases for hexagonal phase irrespective to substrates. From the Fig. 2, the indexed peaks for AlN on Cu substrates at gas mixture ratio of 13:7 are same as the peaks observed from AlN thin film coated over Cu substrates at gas mixture ratio of 16:4 but the intensity of peak observed at 45.50º varies as high for Cu substrates prepared with gas mixture ratio of 13:7. On considering the Al substrates, hexagonal phases are dominated and few cubic phase are observed at low and high N_2 gas mixture ratio. A drastic increment on the intensity of (200) oriented peaks could be observed as with 13:7 ratio gas flow. The peak shifting from lower 2θ to higher 2θ could also be observed for higher N_2 flow. Moreover, unidentified peaks are also observed which are not related to the respective elements or compounds. This may arise as a result of impurity from the substrates. Moreover, a drastic increment on the intensity of hexagonal peak observed at 2θ = 65.08º is also noticed for the AlN thin film coated at 14:6 ratio gas flow. Few cubic phases in addition to the hexagonal phase are also observed when AlN thin film prepared at gas mixed ratio of 13:7. In this case, cubic phase are dominated and hexagonal are suppressed as a result of higher N_2 flow (13:7). Moreover, most of cubic phases exist with Cu substrates with respect to sputtering power and N_2 gas flow ratio. Since, cubic copper substrates enhance the growth of cubic (2 0 0) oriented AlN than h (1 0 3) phase of AlN, mismatch between cubic Cu and cubic AlN thin film are very less and hence suppress of hexagonal AlN growth is observed on Cu substrates [30]. From Fig. 1 and Fig. 2, all samples show very low intensity of (1 0 0) oriented peak when compared to (1 0 3) oriented peak for various sputtering power and N_2 gas flow ratio which exhibit the synthesized film is *c*-axis normal to the substrate [31].

B. *Structural Parameter Analysis*

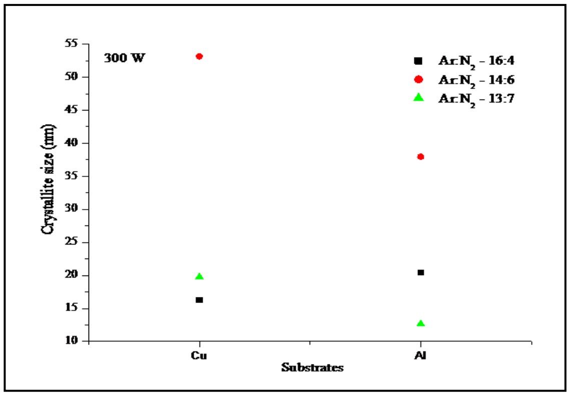
The crystallite size (*D*) was calculated using the Debye Scherer formula [32] from the full width at half-maximum (*w*) measurements:

$$D = 0.94\lambda / w \cos\theta \qquad (1)$$

The crystallite size of all AlN thin film samples are calculated and the observed results for the film prepared at 250 W are plotted in Fig. 3(a) and it reveals that the crystallite size increases for the film prepared over Cu substrates and decreases for Al substrates as N_2 flow ratio increases. Moreover, high N_2 flow does not support on the increase in crystallite size for AlN thin film coated on Cu substrates. Fig. 3(b) shows the results of AlN thin film prepared at 300 W and reveals that the same behavior could be observed as the crystallite size increases with N_2 gas flow ratio increases as observed at 250 W. It is also noticed that the increasing behavior is only for N_2 flow upto 14:6 ratio and decreases noticeably for further increase of N_2 flow. From Fig. 3, high value in crystallite size could be observed for AlN thin film coated over Cu substrates prepared at 300 W sputtering power.



3(a)



3(b)

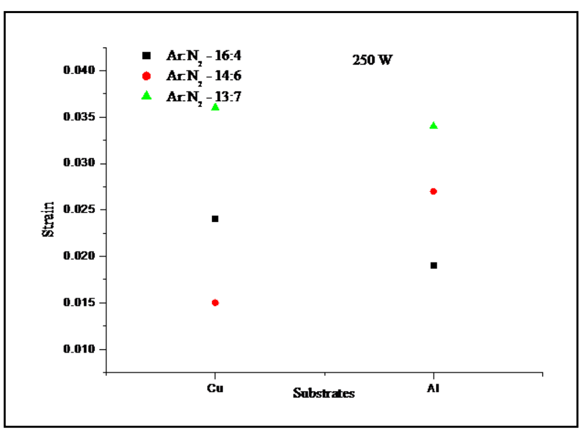
Figure 3 Crystallite size of AlN thin film prepared at (a) 250 W and (b) 300 W sputtering power with various gas flow ratio.

Also, the high sputtering power helps to improve the crystallite size when AlN thin film coated over Al substrates. In addition to this, the dislocation density (δ), defined as the length of dislocation lines per unit volume of the crystal, was evaluated from the relation [33] and the observed results are plotted in Fig. 4(a) and (b).

$$\delta = 1 / D^2 \qquad (2)$$

The strain (ε) was calculated from formula

$$\varepsilon = w \cos \theta / 4 \qquad (3)$$



4(a)

Fig. 4(a) shows the results of AlN thin film prepared at 250 W and explains that an increasing manner in dislocation density is observed for Al substrates as with N_2 flow increases. Even though, high value is observed for AlN prepared over Cu substrates at 250 W power. But very low value in dislocation density could be observed for AlN thin film prepared at 14:6 for both substrates (Cu and Al) when prepared at 300 W (see Fig. 4(b)).

4(b)

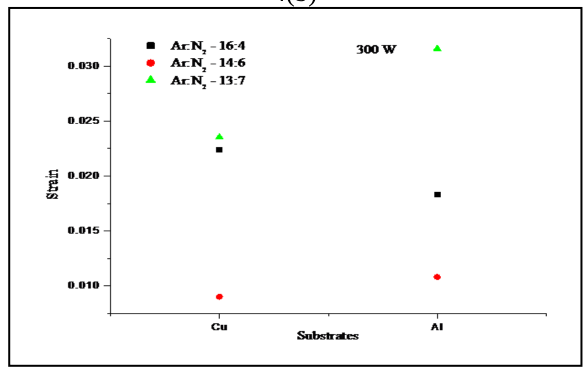
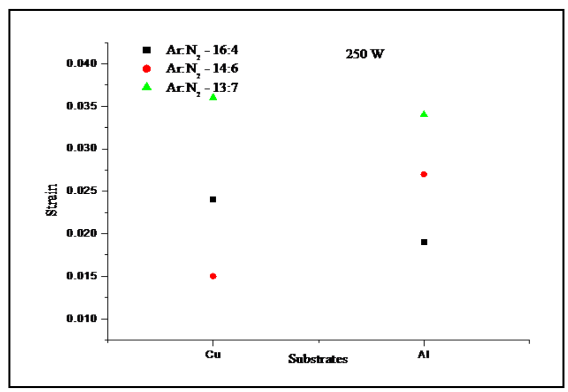
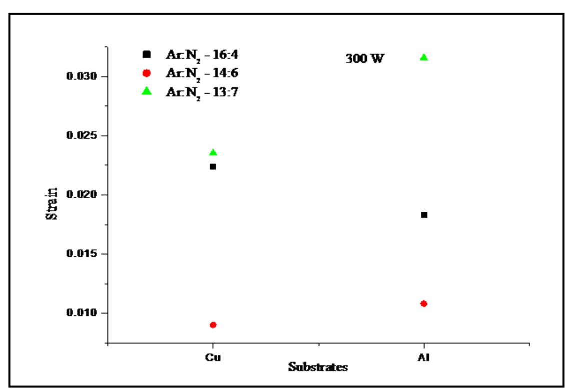


Figure 4 Dislocation density of AlN thin film prepared at (a) 250 W and (b) 300 W sputtering power with various gas flow ratio.

The strain developed during the deposition of AlN thin films over different surfaces are calculated and the observed results are plotted in Fig. 5(a) and (b). As observed for dislocation density, very low value of strain is observed for 300 W for both substrates and the strain increases as the N_2 gas flow increases when the sputtering carried out at 250 W power for AlN on Al substrates.



5(a)



5(b)

Figure 5 Variation in strain during the preparation of AlN thin film at (a) 250 W and (b) 300 W sputtering power with various gas flow ratio.

Noticeable decrease in strain value is observed for AlN thin film prepared over both substrates at 300 W as the N_2 flow increases upto Ar:N_2 - 14:6 ratio. From Fig. 5, substrates and sputtering power influence on increasing the strain value noticeably at high N_2 gas flow ratio.

The internal stress (σ) in the deposited film is calculated using the relation

$$\sigma = - E\,(d_a - d_o) / (2 d_o Y) \quad (4)$$

where d_o and d_a are the d spacing of bulk and thin film forms respectively [34]. E and Y are the Young's modulus and Poisson's ratio of AlN respectively. The Young's modulus and Poisson's ratio of AlN are E = 308 GPa [35] and Y = 0.29 [36] respectively.

The structural parameters are measured from the XRD spectra and given in Table 1 and Table 2. Table 1 shows the results observed form the AlN thin film samples prepared at 300 W at various Ar and N_2 gas ratio. From (3), the nature of stress applied during the growth of crystal could be identified by the sign of the observed value. If the observed value is positive, it represents the compressive stress and if it is negative, the tensile stress is applied during the growth process. From Table 1, tensile stress could be observed mostly for AlN thin film coated over Cu substrates and the applied tensile stress decreases as the N_2 gas flow increases for Cu substrates. Moreover, compressive stress is also observed with Cu substrates at high N_2 flow ratio. But AlN thin film prepared over Al substrates shows compressive nature of stress at high N_2 gas flow ratio.

Table 2 shows the structural properties of AlN thin film prepared at 250 W by varying the Ar and N_2 gas flow ratio. For applied stress based on equation (4), mixed stress (compressive and tensile) could be observed for all samples irrespective to the N_2 gas flow. High value of compressive stress is observed for (200) oriented cubic phase of AlN prepared at Cu substrates. Moreover, the applied stress increases as with N_2 gas flow increases for AlN thin film coated over Al substrates.

Meantime, the stress shows decreasing manner as with N_2 gas flow decreases for Cu substrates. Overall, the applied stress is observed as low for AlN thin film prepared over Cu substrates at low N_2 flow ratio.

C. AFM - Surface Properties

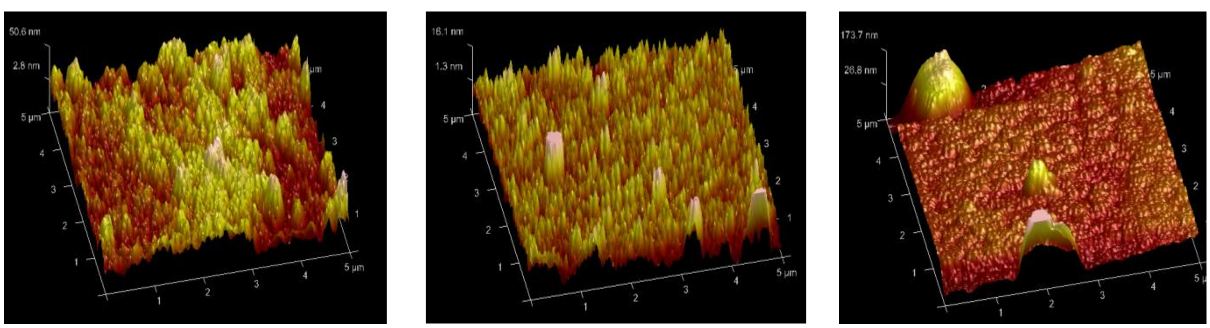
Fig. 6 (a-f) shows the 3D surface morphology of AlN coated Cu and Al substrates prepared at 300 W at various gas flow ratio. The first row (Fig. a, b and c) and second row show the images of AlN thin film coated over Cu and Al substrates respectively. First, second and third columns show the results of various gas flow ratio such as 16:4, 14:6 and 13:7 ratio respectively. It reveals that the surface morphology of AlN thin film reflects the surface of metal substrates as smooth surface for AlN on Cu substrates than Al substrates. The same observation is also observed for AlN thin films prepared at 250 W (see Fig. 7 (a-f)). In order to understand in detail, the surface roughness and the particle size of prepared AlN thin film for various synthesis parameters are recorded and the observed results are tabulated in Table 3. It clearly indicates that the roughness of the prepared film is low for AlN coated over Cu substrates than Al substrates. It could be seen that the roughness is observed as high for 300 W powers at high N_2 flow (13:7).

TABLE I. STRUCTURAL PROPERTIES OF AlN THIN FILM PREPARED OVER DIFFERENT METAL SUBSTRATE AT 250 W FOR DIFFERENT GAS FLOW RATIO

Gas ratio	Substrates	Obs. 2θ	Std. 2θ	Obs. d	Std. d	Hkl	FWHM	Residual stress	JCPDS No.
16:4	Cu	38.98	39.42	2.273	2.284	c111	0.092	0.199289	650841
		45.34	45.84	1.984	1.978	c200	0.11	-0.12552	650841
		66.04	66.06	1.425	1.413	h103	0.118	-0.35142	760702
	Al	40.29	39.61	2.255	2.2736	c111	0.069	0.338521	882250
		58.25	58.86	1.581	1.5675	h110	0.089	-0.35638	700354
		69.57	69.78	1.371	1.3523	h200	0.102	-0.57221	893446
14:6	Cu	39.02	39.42	2.275	2.284	c111	0.023	0.163054	650841
		45.36	45.84	1.983	1.978	c200	0.095	-0.1046	650841
		66.06	66.06	1.415	1.413	h103	0.084	-0.05857	760702
	Al	40.33	39.61	2.254	2.2736	c111	0.177	0.356721	882250
		58.29	58.86	1.582	1.5675	h110	0.084	-0.38278	700354
		69.65	69.78	1.376	1.3523	h200	0.102	-0.72521	893446
13:7	Cu	33.57	33.09	2.693	2.7046	h100	0.063	0.177477	893446
		37.41	37.96	2.377	2.3685	h101	0.278	-0.1485	653409
		38.45	38.26	2.341	2.3502	h101	0.197	0.161983	882360
		42.86	41.81	2.089	2.159	c200	0.072	1.341627	871053
		44.79	45.84	1.992	1.978	c200	0.218	-0.29288	650841
		49.98	49.86	1.811	1.8273	h102	0.054	0.369117	653409
		65.55	65.89	1.427	1.4164	h103	0.225	-0.30968	893446
	Al	37.88	37.96	2.376	2.3685	h101	0.257	-0.13103	653409
		40.25	39.61	2.256	2.2736	c111	0.095	0.320321	882250
		58.21	58.86	1.582	1.5675	h110	0.126	-0.38278	700354
		69.55	69.78	1.379	1.3523	h200	0.115	-0.817	893446

TABLE II. STRUCTURAL PROPERTIES OF AlN THIN FILM PREPARED OVER DIFFERENT METAL SUBSTRATE AT 300 W FOR DIFFERENT GAS FLOW RATIO

Gas ratio		Obs. 2θ	Std. 2θ	Obs. d	Std. d	hkl	FWHM	Residual stress	JCPDS No.
16:4	Cu	39.03	39.42	2.3046	2.284	c111	0.088	-0.37321	650841
		45.31	45.84	1.99622	1.978	c200	0.107	-0.38116	650841
		66.06	66.06	1.4126	1.4033	h103	0.104	-0.27423	760702
	Al	38.50	38.26	2.3350	2.3502	h101	0.082	0.267624	882360
		40.25	39.61	2.2384	2.2736	c111	0.079	0.276641	882250
		58.22	58.86	1.5835	1.5675	h110	0.092	-0.42238	700354
		65.31	65.89	1.4352	1.4164	h103	0.06	-0.54924	893446
		69.54	69.78	1.3504	1.3523	h200	0.102	0.058139	893446
14:6	Cu	33.04	33.09	2.7099	2.7046	h100	0.056	-0.08109	893556
		59.16	59.12	1.5724	1.5615	h110	0.021	-0.28885	893446
	Al	33.14	33.09	2.70996	2.7046	h100	0.063	-0.08201	893556
		38.50	38.26	2.3167	2.3502	h101	0.027	0.589829	882360
		65.08	65.89	1.4233	1.4164	h103	0.052	-0.20158	893446
13:7	Cu	39.19	39.42	2.3056	2.284	c111	0.202	-0.39133	650841
		45.50	45.84	1.925	1.978	c200	0.123	1.108756	650841
		66.18	66.06	1.4126	1.4033	h103	0.081	-0.27423	760702
	Al	33.28	33.24	2.6892	2.6933	h101	0.108	0.062992	653409
		38.59	38.26	2.3358	2.3502	h101	0.202	0.253539	882360
		40.31	39.61	2.2384	2.2736	c111	0.079	0.640641	882250
		58.29	58.86	1.5835	1.5675	h110	0.092	-0.42238	700354
		65.20	65.89	1.4352	1.4164	h103	0.06	-0.54924	653409
		69.61	69.78	1.3511	1.3466	h200	0.084	-0.13828	653409



(a) (b) (c)

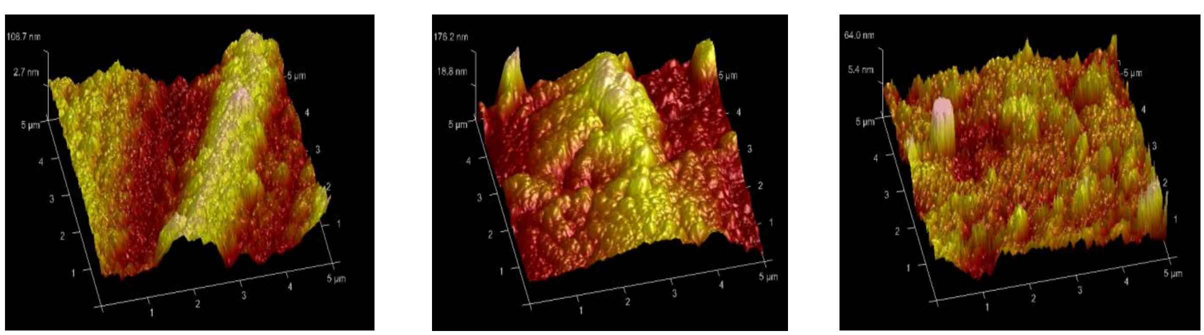
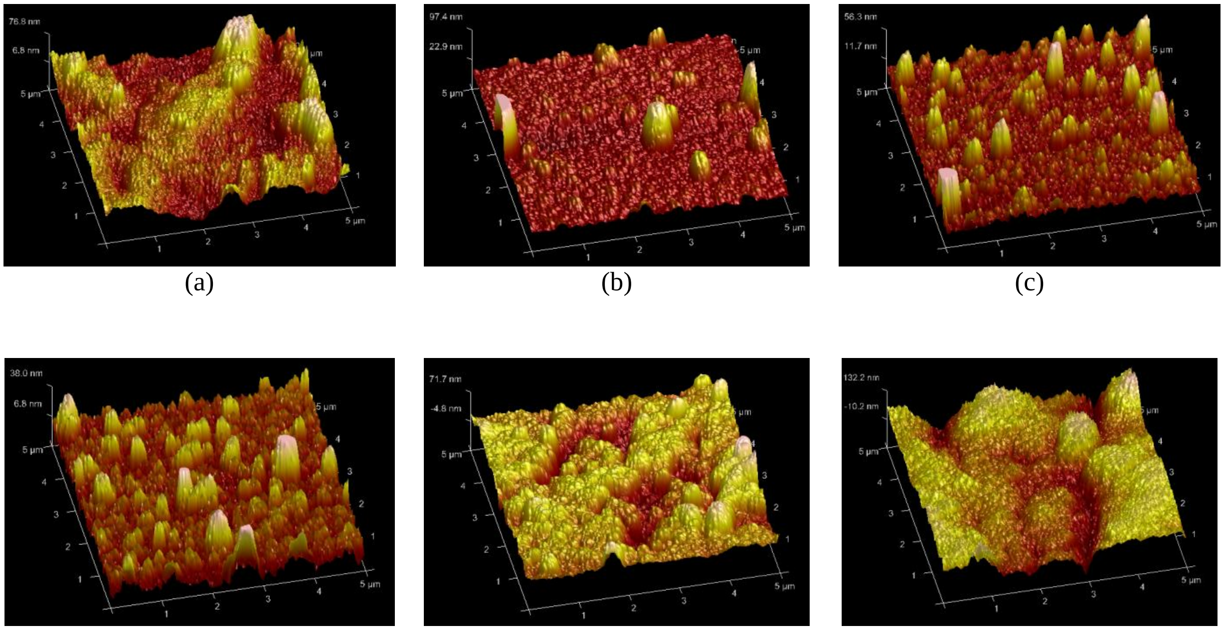


Figure 6 AFM images of AlN thin film prepared at 300 W sputtering power (Row 1 – Cu substrate and Row 2 – Al substrate, Column (a & d) 16:4, (b and e) 14:6 and (c and f) 13:7)



(a) (b) (c)

(d) (e) (f)

Figure 7 AFM images of AlN thin film prepared at 250 W sputtering power (Row 1 – Cu substrate and Row 2 – Al substrate, Column (a & d) 16:4, (b & e) 14:6 and (c & f) 13:7)

High value is observed for Al substrates as 98 nm. Noticeable results are observed with Cu substrates as the roughness decreases as the sputtering power increases from 250 to 300 W irrespective to the gas flow ratio. This may be attributed to the influence of applied stress during the growth of AlN on Cu substrates at this N_2 flow rates. In addition, the particle size of the AlN thin film samples are also measured by processing the AFM image and given in Table 4. It shows that very big particles (546 nm) are observed for high N_2 flow at 250 W power. From Table 4, an increment in particle size for applied sputtering power could be observed as the N_2 gas flow increases. Overall, the sputtering power and gas flow influences the surface morphology as well as the change in particle size when AlN thin film prepared on Cu and Al substrates.

TABLE III. SURFACE ROUGHNESS OF AlN THIN FILM FOR VARIOUS SYNTHESIS PARAMETER

Roughness (nm)	**Cu**			**Al**		
	Gas ratio			Gas ratio		
	16:4	14:6	13:7	16:4	14:6	13:7
300 W	12	12	16	30	38	98
250 W	15	23	17	27	15	33

TABLE IV. PARTICLE SIZE OF AlN THIN FILM FOR VARIOUS SYNTHESIS PARAMETERS

Particle size (nm)	**Cu**			**Al**		
	Gas ratio			Gas ratio		
	16:4	14:6	13:7	16:4	14:6	13:7
300 W	175	206	341	163	202	283
250 W	217	245	302	129	241	564

Cu substrate supports the growth of cubic phases at high N_2 flow with 300 W sputtering powers. At high N_2 flow rate (13:7), the possibility in the formation of cubic AlN phase is high as with sputtering power increases from 200 to 300 W. The intensity of the peaks shifted towards higher 2θ as with gas flow and sputtering power increases. Structural parameters such as crystalline size, dislocation density and strain were affected by the higher N_2 gas flow ratio than the sputtering power. AlN on Cu substrates showed smoother surface than Al substrates and also high surface roughness was also observed with high N_2 gas flow. The sputtering power and gas flow influenced the change in particle size with respect to metal substrates.

ACKNOWLEDGMENT

The authors would like to thank the Nano Optoelectronics Research Laboratory in School of Physics where all AlN thin film samples were synthesized by using DC sputtering system and the XRD lab for providing the crystallographic analysis facilities.

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